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**REPRESENTATIVENESS OF RESIN
SAMPLES OBTAINED WITH THE
RESEARCH DIVISION RESIN SLURRY SAMPLER**

Report No 87-34-K

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Engineer - Water Research
Process Chemistry Section
Chemical Research Department

ABSTRACT

Tests were carried out to determine the representativeness of resin samples collected with the Research Division Resin Slurry Sampler during resin slurry transfers. Resin samples can be collected without interfering with slurry transfer. A bias in sample composition was detected favouring resin from the bottom of the service column. It is recommended that the Resin Slurry Sampler be used when semi-quantitative information on resin composition is acceptable. Observations on resin slurry transfer performance were made during this study and are also reported.

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EXECUTIVE SUMMARY

REPRESENTATIVENESS OF RESIN SAMPLES OBTAINED
WITH THE RESEARCH DIVISION RESIN SLURRY SAMPLER

D.H. Barber
Process Chemistry Section
Chemical Research Department

The Research Division Resin Slurry Sampler was developed in 1982 to obtain samples of resin during slurry transfer of active resins. At that time the relationship between the compositions of the sample and the resin transferred was not determined. Resin beds containing layers of different ionic forms of cation resin were manually loaded into an ion exchange column. The IX column was a replica of a seven cubic foot Bruce NGS moderator IX column. The resin was sampled during a slurry transfer to a second column. The composition of the sample was compared with the initial bed. A bias was detected in the samples favouring resin from the bottom of the column. It is recommended that the Resin Slurry Sampler be used when semi-quantitative information on resin composition is acceptable.

During this study and a sampler demonstration programme observations on methods of improving the reliability of slurry transfers were made. These observations, which should be useful in the design and operation of resin slurry transfer systems, are also reported.

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To Mr. R.T. Popple
Manager
Radioactivity Management and
Environmental Protection Department

Attn: Mr. I. Benovich

**REPRESENTATIVENESS OF RESIN SAMPLES OBTAINED
WITH THE RESEARCH DIVISION RESIN SLURRY SAMPLER**

1.0 INTRODUCTION

The resin slurry sampler shown in Figure 1 was developed by the Chemical Research Department (CRD) of Ontario Hydro Research Division in 1982./1/ The sampler was intended to be used to collect small samples of active resin for detailed analysis. The analysis could be for either active waste characterization or for operational troubleshooting. The resin slurry sampler was used at NPD to collect samples from CAN-DECON resin columns/2/ and at Douglas Point/3/. The question of sample representativeness was not assessed in the initial sampler development programme.

The objective of the current test programme was to determine the representativeness of resin samples obtained with the sampler to allow potential users of the sampler to assess the adequacy of the samples for their purposes. The test programme was requested by Radioactivity Management and Environmental Protection Department (RMEP) and co-funded by RMEP and CRD.

During the course of this programme and a parallel programme of sampler demonstration to station personnel, a number of observations on slurry transfer behaviour were made. These observations are recorded here for completeness.

2.0 EXPERIMENTAL SET-UP

Resin beds were prepared by manually loading different amounts of resin in different chemical forms into the test column (Column T in Figure 2) in a layered arrangement. The layering allowed determination of whether a bias in the sampling favoured the top or bottom of the original resin bed. Resins in potassium form and zinc form were prepared in the Research Division's Ion Exchange Pilot Plant. The hydrogen-form, chloride-form and sodium-form resins were bought in those forms. The resin was transferred in a water slurry to the receiving column with the slurry sampler installed as shown in Figure 2.

Slurry water flow rates were measured with a flowmeter on the downstream side of the receiving column. It was intended to run three experiments at each of two flow rates. The slurry water flow rates were 12.5 L/min and 25 L/min.

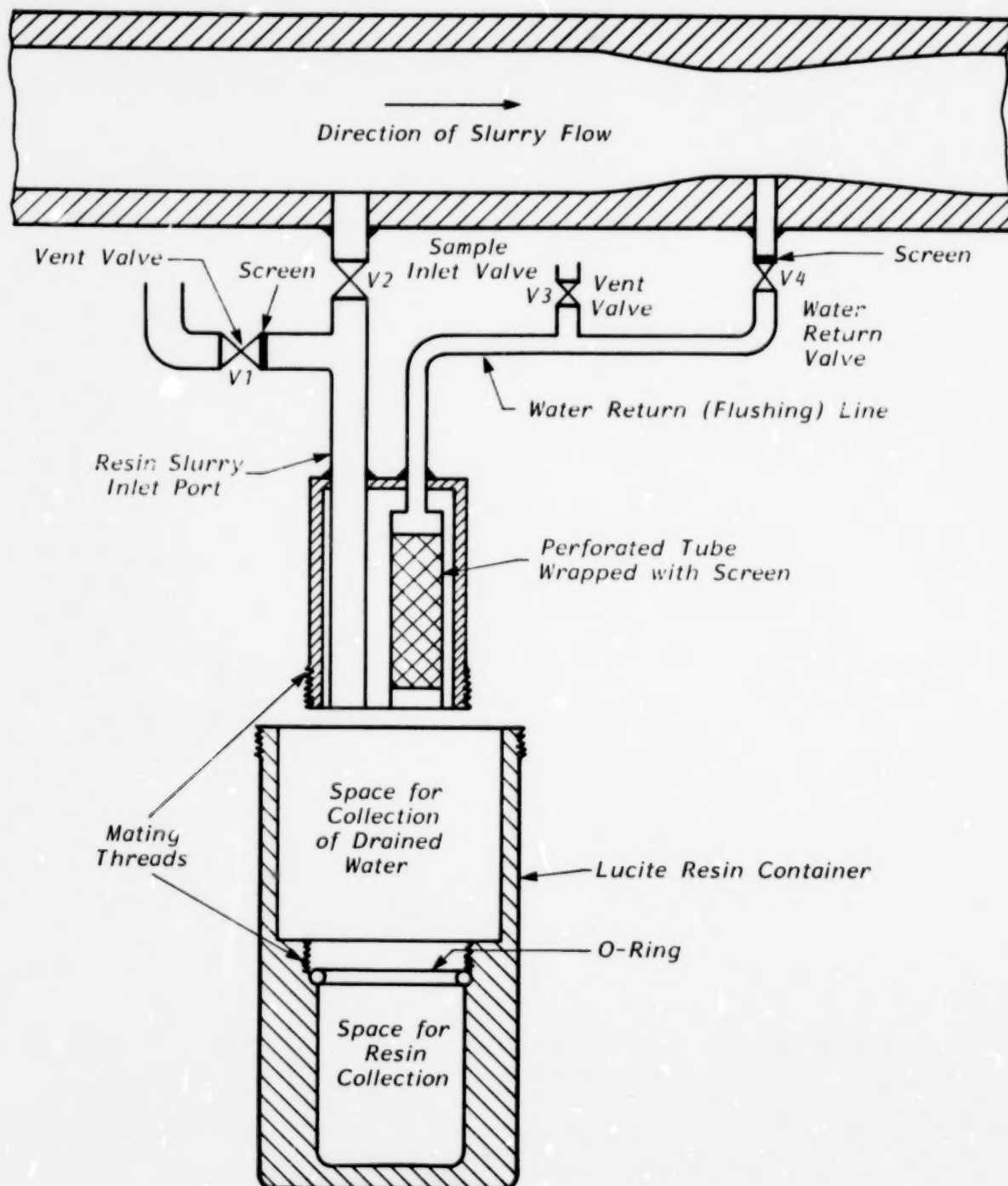


FIGURE 1
SKETCH OF RESIN SAMPLER
(NOT TO SCALE)

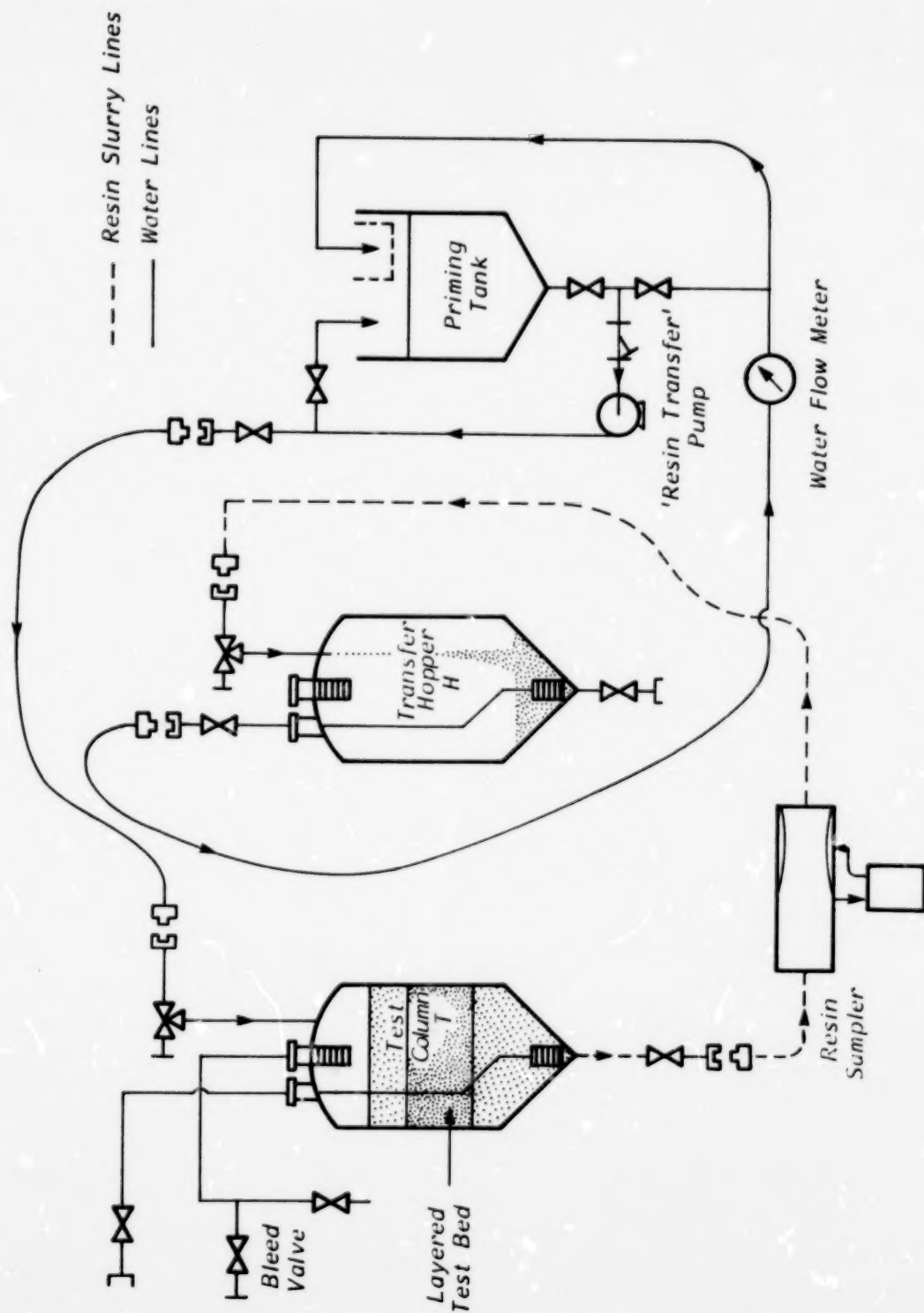


FIGURE 2
SCHEMATIC OF SET-UP FOR TESTING OF RESIN SAMPLER

3.0 SAMPLER OPERATION

The direction of resin slurry flow is indicated by the arrow (Figure 1). At the start of the slurry transfer, the vent valves (V1 and V3) were closed. The sampling valve (V2) was open. The resin slurry lines and the sample container were filled with water. At the high flow rate (25 L/min), the water return valve (V4) was closed. With that valve (V4) open sample collection would be rapid and would overflow the container. At the lower flow rate (12.5 L/min), the water return valve (V4) was open.

Because of the venturi there is a lower pressure above the water return valve (V4) than above the sampling valve (V2). Opening the water return valve (V4) allows the pressure difference to force some of the resin slurry water through the sample collector. The resin is trapped in the sample container by the cylindrical screen on the water return line. Opening the water return valve (V4) could be used to start sample collection at the higher flow rate (25 L/min). Valve (V4) would be closed once sample flow had been established.

4.0 ANALYSIS

A sample of known volume of each metal-form cation resin was regenerated with an excess of 2N hydrochloric acid. The effluent solution was analyzed by atomic absorption spectroscopy for the metal of interest. Initial resin loadings were calculated for each metal form.

The samples collected with the slurry sampler were also regenerated with hydrochloric acid and strip solutions were analyzed by atomic absorption spectroscopy for sodium, potassium and zinc. The amount of each form of resin in the sample was calculated from these analyses and the initial resin loadings.

5.0 RESULTS

A total of five runs were made. Four were made at the high (25 L/min) slurry water flow rate. One run was made at the low (12.5 L/min) flow rate. The initial bed contents and results of the resin sample analyses for the two acceptable runs are reported in Table 1. The observations show a slight bias in the samples. Both samples appear to be rich in the resin forms from the bottom of the test column. The runs reported were both performed at the high slurry water flow rate (25 L/min).

In the case of Run A1, the resin sample as initially collected showed stratification into three layers of two shades of resin corresponding to the layering of the initial bed. Unfortunately, the layering was disturbed on removing the sample container from the sampler.

During Run A2, operator intervention to open valve V4 was required to start sample collection. This valve was subsequently closed. Near the end of the run pulsation of the slurry line was experienced.

Three runs were made with unsatisfactory results. In the first, a mixed chloride-form anion, hydrogen-form cation and sodium-form cation resin was used. The slurry water flow rate was uncontrollable due to clumping of the chloride-form anion resin and the hydrogen-form cation resin. The samples collected during these runs were not analyzed.

Table 1

Layer	Ionic Form	Volume in Bed (ft ³)	% of Bed Volume	% of Sample Volume	% of <u>Sample</u> % of Bed
Run A1					
Top	H ⁺	2	28.6	12	0.41
Middle	Na ⁺	2	28.6	26	0.91
Bottom	K ⁺	3	42.9	62	1.45
Run A2					
Top	K ⁺	2	28.6	11	0.38
Middle 1	H ⁺	2	28.6	37	1.29
Middle 2	Na ⁺	2	28.6	34	1.19
Bottom	Zn ⁺⁺	1	14.3	19	1.33

The second unsuccessful run was made at the low flow rate (12.5 L/min) with cation forms of resin. Initially there was no sample collection with valve V4 closed. Sample collection was possible with valve V4 open. However, at the low flow rate pulsation led to plugging of slurry lines; intermittent, but rapid, sample collection; and premature filling of the sample container.

The third unsuccessful run was made with four cation-form cation resins at the high (25 L/min) slurry water flow rate. This slurry was also subject to pulsation and intermittent, rapid sample collection.

6.0 ADDITIONAL OBSERVATIONS

The following observations were made during this programme and a parallel programme of sampler demonstration to station personnel and have already formed the basis for recommendations regarding resin slurry transfer operation. They are reported here for completeness.

During some demonstration, calibration and commissioning runs a small flow (about 2 L/min) of water was introduced into the slurry outlet line of the service column. During these runs there was no pulsation during the slurry and pressures in the slurry loop were lower than during other runs. During one run, pulsations, which had started during the slurry, stopped shortly after starting this flow. Normally following the onset of pulsations, the pulsations remain until either the slurry is over or flow stops.

During a demonstration using 3/4 inch hose to simulate Bruce NGS-A conditions, the slurry water was added through the purification outlet screen. This practice has been adopted at

Bruce NGS-A to avoid plugging problems. During this run, no pulsation was observed and sample collection was smooth and continuous.

7.0 DISCUSSION

From the observations reported above it is clear that the Resin Slurry Sampler can be used to collect resin samples during a slurry transfer and that given a smooth transfer at slurry water flow rates of about 25 L/min, the sample bears a reasonable resemblance to the original bed. The bias in the results means that concentrations of strongly held ions that would be expected to be associated with the top of the resin bed may be under-estimated and that concentrations of less strongly held ions (from the bottom of the bed) might be overestimated. It is also apparent from the qualitative observations reported above that a single slurry transfer of the resin from a station service column to a dedeuteration hopper or transfer hopper cannot be expected to thoroughly homogenize the spent resin bed. Back-washing of the resin in the transfer hopper or dedeuteration hopper could partition the resin by particle size and density, and could be expected to change but not eliminate the bias. In either case, a bias can be expected and this should be considered when resin analyses are used for assessment or design purposes.

The question of what is an acceptable bias in a sample depends on the purpose of the sampling and subsequent analysis. In some situations the property of interest could be independent of position and any sample which can be safely collected is of value. In other situations involving mass balances the sampling bias might be a major contribution to the error in analysis results. It should, therefore, be the end use of the resin analysis which determines whether collection of samples with the slurry sampler meets the requirements.

The smoothness of a slurry operation has a large role in the success of both the slurry transfer and slurry sampling. It is unclear why pulsations start during some transfer but not during others under similar conditions. Plugging of moderator purification resin transfer lines in certain circumstances could have adverse impact on purification and station availability. Since the pulsations have an effect on the reliability of slurry transfer operations, station personnel have found ways to overcome them. At Bruce NGS-A during transfer from the resin chariot to the dedeuteration tanks, the slurry water is introduced through the bottom screen of the column from which resin is being transferred. This practice undercuts the resin bed and should result in a slurry of lower solids content than adding slurry water to the top of the vessel. During the tests for sample representativeness the bypass flow was not established. The test conditions were therefore a worst case as far as obtaining a smooth slurry is concerned.

8.0 CONCLUSIONS

1. Samples collected in the absence of pulsation of slurry flow contain slightly more resin from the bottom of the service column than from the top. Analysis of these samples would provide a means of obtaining a semi-quantitative analysis of the resin slurried out of a service IX column.
2. At slurry water flow rates of 25 L/min or greater valve V4 on the water return line of the sampler should be closed during slurring. At flow rates of 12 L/min or less valve V4 should be open during slurring.

3. Pulsation in the resin transfer lines lead to fast sampling rates and can lead to filling of the sample container before the end of the slurry transfer.
4. Sampler operation does not adversely affect slurry transfer operations.
5. A small bypass flow of water around the column from which resin is being transferred reduces pulsation and improves transfer reliability. This precaution decreases the slurry solids content during resin transfer and allows a more reliable slurry transfer. This flow should be maintained whether or not slurry sampling is being done.

9.0 RECOMMENDATIONS

1. It is recommended that the Research Division Resin Slurry Sampler be used when a semi-quantitative analysis of an active resin sample is acceptable. The sampler can be employed during slurry transfers between IX columns, dedeuteration tanks or transfer hoppers.

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REFERENCES

1. Nott, B.R. "Resin Slurry Sampler Development," Ontario Hydro Research Division Report No. 82-147-H, Toronto, Ontario, (April 13, 1982).
2. Chartier, D.R. "NPD CANDECON Resin Analysis," Ontario Hydro Research Division Report No 86-3-H, Toronto, Ontario, (February 24, 1986).
3. Chartier, D.R. "Analysis of a Resin Sample from Douglas Point NGS," Ontario Hydro Research Division Report No. C84-13-K, Toronto, Ontario, (February 1, 1984).

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